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(54) Coated cosmetic materials and method of coating cosmetic materials.

(57) An improved coating for cosmetic materials is disclosed. Fluoroalkylpolysiloxanes used as coatings for cosmetic powders and microparticulate materials provides the advantages of existing polysiloxane coatings plus provides advantages making the coated materials useful in emulsion systems and alkaline systems even when aged at high temperatures.

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The present invention relates to the use of trifluoroalkyl polysiloxane compounds as coating for powders and particulates, in particular to cosmetic materials and improved cosmetic formulations resulting from coating applied to powders and particulates used in cosmetic formulations.

BACKGROUND

The increasing importance of personal appearance, whether in projecting a healthy, youthful or stylish image, has led to an increasing demand for cosmetics. The demand is being met by a growing number of products and suppliers with an attendant expansion in competition.

Product quality is important and is reflected by many qualities and characteristics of each product. Physical appearance of the product should be homogeneous. Any streaking, settling or separation has an adverse impact on the consumer.

A cosmetic should apply smoothly, have a good skin adhesion and a good feel. A makeup that applies unevenly, flakes, smears or feels overly dry or oily is not likely to be favorably received.

Color consistency from batch to batch is also of importance. Small variances in shade are noticeable in cosmetics and accordingly a consumer's expectation in receiving the same shade as previously obtained should be fulfilled.

Cosmetics use varying amounts of particulates including pigments, pearlescent materials, extenders, etc., dispersed in oils, waxes and emulsions. Uniform dispersion, suspension stability and particulate loading in a suspension are all factors affecting the product quality. Obviously, optimization of all is desirable.

Problems are encountered in trying to improve one or two factors without adversely affecting another or improving desired characteristics without introducing other detrimental effects.

Coating of pigments, extenders and fillers has been used to enhance dispersion of particulates by increasing hydrophobicity. Increased hydrophobicity also aids in the suspension of particulates in cosmetic preparations.

Pigments have been treated with lecithin along with lecithin component fatty acids and related fatty acids. Metal alkoxides, metal soaps and metal salts of fatty acids have been used with success.

Silicone treatment of pigments is another method of treatment and results in improved hydrophobicity. Dispersion, suspension and loading characteristics are all improved as are the water repellence and spreadability of a product incorporating the treated pigment. Enhanced skin adhesion and reduced color change on skin are also benefits of silicone treatment.

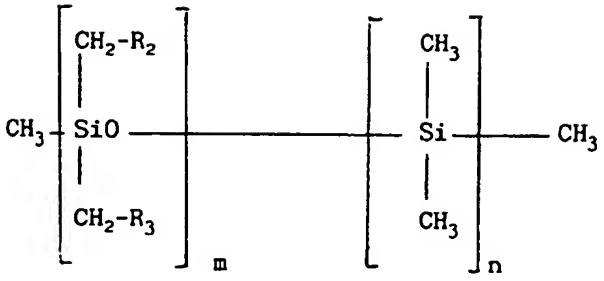
Simethicone (dimethyl polysiloxane) has been widely used as a coating and is one of the simplest, structurally, of the silicones used. However, with all the advantages associated with silicone treatment, a problem common to all the silicones results in adverse characteristics in the final product, including the failure to have extended shelf life.

SUMMARY OF THE INVENTION

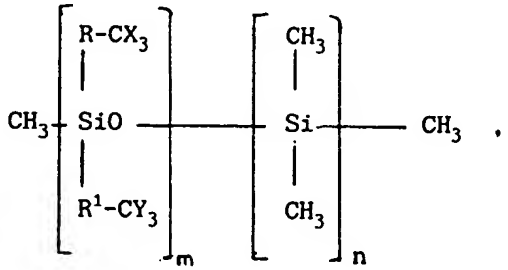
Hydrogen generation has been observed from the silicone treated pigments and the hydrogen generation may be pronounced in emulsion systems and alkaline systems. The hydrogen generation is severe enough to raise questions about whether the silicone treatment is desirable in such systems or where a long shelf life is required.

The invention is intended to provide a remedy to this problem. It solves the problem of how to achieve desired results in optimizing cosmetic characteristics without introducing some of the known drawbacks of existing formulations.

The invention provides a powder or particulate material coated with a trifluoroalkylpolysiloxane compound of the general formula



wherein m is an integer averaging from 1 to 1000;
n is 0 or 1; and
R_2 and R_3 is each hydrogen or an alkyl group of from 1 to 9 carbon atoms;
wherein at least one of R_2 and R_3 is a fluoroalkyl group substituted with from 1 to 5 fluorine atoms and having at least two carbon atoms if substituted with four or five fluorine atoms;
and wherein said fluoroalkyl groups are randomly or uniformly distributed in the polysiloxane structure, such as a formula

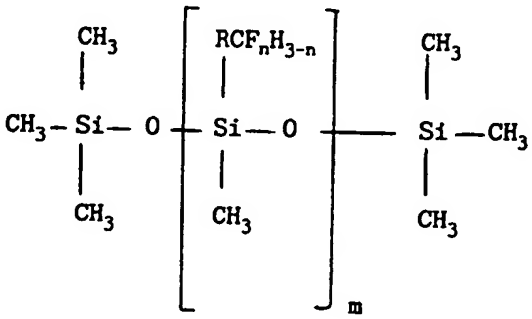


wherein
m is an integer averaging from 1 to 1000;
n is 0 or 1;
R and R^1 are alkyl groups of from 1 to 10 carbons;
X is hydrogen or fluorine;
Y is hydrogen or fluorine where at least one of X and Y is fluorine; and the trifluoro groups may be randomly or uniformyl disposed over the polysiloxane structure. The powder or particulate material is very well suited for use in cosmetic products. Preferably, the particulate material is a microparticulate material.

As explained below it is preferred that the coating comprises 0.01 to 30.0 wt.% of the coated material.

Preferably, the above-mentioned fluoroalkyl group includes a trifluoromethyl group.

In a specific embodiment the present invention provides a powder or particulate material coated with a fluoroalkyl polysiloxane compound of the general formula



m is an integer averaging from 1 to 500;
n is 0, 1, 2 or 3;

R is an alkyl group of 0 to 10 carbons;
and the fluoro groups may be randomly or uniformly disposed over the polysiloxane structure.

The invention also relates to cosmetic compositions comprising a modified powder or particulate material, especially a micro-particulate material, as defined in the above. Such a cosmetic composition may preferably be an aqueous cosmetic dispersion containing a cosmetic powder.

In general, the invention relates to the use of trifluoroalkylpolysiloxane compounds as defined in the above for coating powders or particulate materials, which may be suitable for use in cosmetic products. The term "cosmetic" also involves similar uses such as other uses on the skin or body of mammals including human beings. Silicone treatment achieves many desired advantages for incorporating pigments and other cosmetic materials in cosmetic products. Certain silicone structures have been modified and result in coatings that are useful without the drawback of hydrogen production.

In principle, substitution for the methyl units in simethicone will retain the silicone-oxygen backbone of the silicone structure. The beneficial effects as a coating can also be retained. However, the substitution of hydrogen or an alkyl group for the methyl group does not improve the hydrogen generation problem.

Moreover, substitution of a non-halogen, other than hydrogen or an alkyl group, for the methyl group can adversely affect the hydrophobicity of the compound.

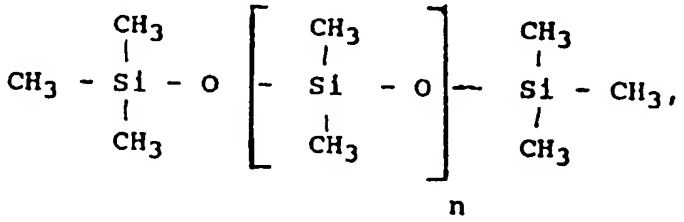
Substitution with halogens can preserve the hydrophobicity but may also produce diatomic halogen gas or a gaseous hydrogen halide acid in addition to the hydrogen gas. Flourine has, however, in some instances been found to behave differently in this respect from the other halogens.

In accordance with the present invention, it has been found that fluorine combined with carbon in a terminal methyl group extending from, but not directly bonded to, a silicone atom has been found to produce an extremely stable compound. More particularly, fluoroalkyl-, difluoroalkyl-and trifluoroalkyl-polysiloxanes, in accordance with the invention, all show great stability and avoid the problem of hydrogen generation. Fluoroalkyl- polysiloxanes where the fluorine is on a terminal methyl group of up to a 10 carbon alkyl group bonded to a silicone atom are also effective. Fluorine may be present on more than one methyl group as long as the methyl group bonded to the silicone backbone is not fluorine bearing.

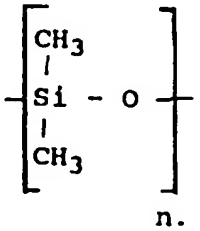
Trifluoroalkylpolysiloxanes are commercially available from Grant Industries, Inc. under the name Gransil and have proved most satisfactory in coating pigments, extenders, pearlescents and filler material for cosmetics.

It is also been discovered by experimentation that the fluoroalkyl groups can be uniformly or randomly spread about the silicone-oxygen backbone. The fluoroalkyl groups discourage hydrogen generation when as few as approximately one fluoroalkyl group per ten silicone-oxygen units is present.

Dimethicone is represented by the formula:

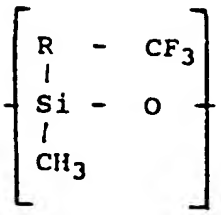


or even
more basically as:

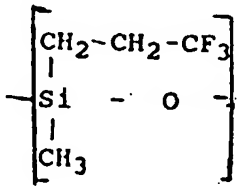


Substitution of a fluorinated alkyl group of 2 to 10 carbons (-R-CF_3) for some methyl (-CH_3) groups in

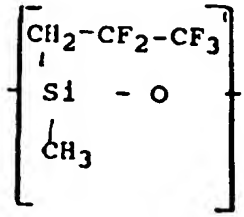
dimethicone to yield units of



achieve the desired result. The terminal fluorinated methyl group may be $-CH_2F$, $-CHF_2$ or $-CF_3$. Methyl ($-CH_2-$) units in the alkyl group R may also be fl rinated as long as the initial methyl unit, that unit bonded to the silicone, is not fluorinated. For example, polysiloxanes having units of



and others having



interspersed along the basic dimethicone structure have both proved very suitable.

The fluoroalkyl-polysiloxane may average up to 500 or 1000 monomer units in length although units in the 1 to 500 range are preferred. Sub ranges of 1 to 100 and 100 to 500 are both very effective.

Alkyl units up to 10 carbons in length may be bonded to a silicone atom although 2 to 5 carbon alkyls are preferable.

The inventive fluoroalkylpolysiloxanes have beneficial effects as a cosmetic particulate coating when as little as 0.01 weight percent of the coating, with respect to the coated particulate, is used. In excess of 30.0 weight percent of the coating may be used although such great amounts are not necessary.

Fluoroalkylpolysiloxanes and products incorporating the inventive coating have a high degree of solvent resistance and retain their excellent stability even when aged at temperatures as high as 45°C.

Surface treatment, in accordance with the invention of pigments, pigment extenders and particulate matter used for decorative and non-decorative cosmetics using substituted fluoroalkylpolysiloxanes results in enhanced lubricity and water repellency (hydrophobicity) and greater adhesion to the skin with no tactile negatives. When dispersed with other materials it gives less color change on the skin and with emulsion systems these surface treated materials are suspended easily and are completely stable even under alkaline conditions.

Treatment of pigments, pigment extenders and other particulate matter may be achieved by the following method in accordance with the invention.

This method of treatment comprises the addition of 0.01 to 30 weight percent (typically found to work well in the range of about two percent) of the fluoroalkylpolysiloxane to the solid material (pigment, pigment extender or other particulate matter) by spraying into a fluidized, agitated filler bed or prilling tower containing the cosmetic raw material. The sprayed powder is then transferred to a mixer such as a PK twin shell blender (with intensifier bar). One may also use a Littleford-Lodige mixer granulator, a ribbon blender,

pan mixer, paddle mixer, a vertical screw mixer, turbine mixer, twin rotator mixer or Muller mixer. The mixing is continued in the PK blender mix until adequate treatment is achieved. This may be determined by the degree to which the solid particulates, with their various porosities have absorbed the fluoroalkyl-polysiloxanes.

The treated material is then pulverized by using a suitable micropulverizer. One may also employ a hammer mill, cage mill, tumbling ball mill, roller mill, disc mill, fluid energy mill or any suitable micronizer. The milling or pulverizing process is repeated usually 2 to 3 times to obtain uniform and desired particle size. Desired particle size is selected in accordance with the size typically and conventionally required for the particular end product being manufactured.

The treated powders/particulate matter may be composed of any of the following powdered materials as single ingredients or combinations thereof:

Pigments:

Organic Colors
Titanium Dioxide
Zinc Oxide
Iron Oxides (Red, Black & Yellow)
Zirconium Oxide
Ultramarine (Blue, Violet & Pink)
Prussian Blue
Chromium Oxides
Chromium Hydroxides
Manganese Violet
Carmine
Ferric Ferrocyanides
Ferric Ammonium Ferrocyanides
Iron Hydroxides

Pigment Extenders:

Talc
Kaolin
Magnesium Carbonate
Calcium Carbonate
Boron Nitride
Sericites
Mica
Aluminium Hydroxide
Bismuth Oxychloride
Magnesium Aluminum Silicate
Silica Beads
Aluminum Silicate

Other Particulate Matter:

Nylons (Polyamides)
Sunscreens
Cellulose
Ceramic Beads
Polymethacrylate polymers and copolymers
Ethylene/Acrylates polymers and copolymers
Styrene/Divinylbenzene polymer and copolymers.

The treated powders are then utilized to prepare various cosmetic formulas for such products as eye Shadows, blushers, face powders, lipsticks, mascara, liquid eyelines, cream make-up, liquid make-up, liquid eye shadow, nail polish, treatment skin lotions and creams, multiphase emulsions, lip gloss, eye pencils, lip

pencils and rouge.

Four typical make-up formulations using materials coated with the fluorinated polysiloxane are given below. It is noted that some ingredients may be totally dispensed with, additional ingredients may be used and most if not all ingredients may be substituted for.

Example 1-1

A typical eye shadow makeup products were prepared using the indicated weight percent of various ingredients. Fluoroalkylpolysiloxane treated materials are noted as "treated in the table below.

Material	Weight Percent Range	Preferred Composition
Talc	70 to 80	74.7
Zinc Stearate	3 to 9	6.0
Magnesium Carbonate	0 to 5	2.0
Treated Titanium Dixoide	1 to 5	2.0
Treated Iron Oxides	0 to 3	1.0
Treated Ultramarine Blue	1 to 15	8.0
Squalane	0 to 10	4.0
Mineral Oil	0 to 5	1.0
Lanolin Alcohol	0 to 3	0.5
Preservatives	0 to 3	0.8

The manufacturing process comprises mixing the powder ingredients, followed by addition of the liquid/oil phase and mixing well. The resultant mixture was then pressed into a mold. Suitable results may be achieved by varying ingredients within the ranges indicated above.

Example 2-1

A typical blusher makeup product is prepared using the weight percent of the various ingredients indicated below, including fluoroalkylpolysiloxane treated materials.

Material Weight	Percent Range Range	Preferred Composition
Treated Talc	40 to 60	47.7
Bismuth Oxychloride	3 to 15	10.0
Mica	5 to 20	18.0
Zinc Stearate	0 to 10	4.0
Treated Titanium Dioxide	3 to 15	10.0
Treated Iron Oxides	1 to 5	2.0
Treated D&C Red 30 Al Lake	0 to 3	0.5
Treated Ultramarine Blue	0 to 3	0.5
Squalane	0 to 10	4.0
Mineral Oil	0 to 5	1.0
Isopropyl Palmitate	0 to 5	1.5
Preservatives	0 to 3	0.8

The manufacturing process comprises mixing the powder ingredients, following by addition of the liquid/oil phase and mixing well. The resultant mixture was then pressed into a mold. Suitable results may be achieved by varying ingredients within the ranges indicated above.

Example 3-1

The lipstick formula prepared using treated cosmetic materials is shown in example 3-1 using the weight percent of various ingredients, including fluoroalkylpolysiloxane treated materials.

Material	Weight Percent Range Range	Preferred Composition
Triisocetyl Citrate	40 to 60	51.8
Ozokerite	0 to 10	6.0
Beeswax	0 to 10	6.0
Carnauba Wax	0 to 5	2.2
Candelilla Wax	0 to 5	1.0
Paraffin	0 to 5	1.5
Lanolin Alcohol	0 to 5	2.0
Castor Oil	5 to 15	11.9
Treated Nylon-12	0 to 5	2.0
Treated D&C Red 6 Ba Lake	1 to 3	1.6
Treated D&C Red 7 Ca Lake	1 to 3	1.7
Treated Iron Oxides	0 to 3	0.6
Treated Bismuth Oxychloride	0 to 2	0.4
BHT	0 to 2	0.5
Preservatives	0 to 2	0.3
Perfume	0 to 2	0.5

The waxes and oils are heated to 85° C. The colors, which have been roller milled previously in castor oil blends, are added slowly and the mix is blended all together at 85° C until uniform then cooled to 70° C and molded.

Example 4-1

A waterproof mascara product was prepared using the weight percent of various ingredients.

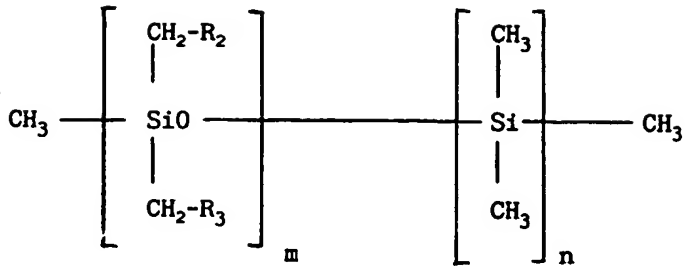
Material	Weight Percent Range Range	Preferred Composition
Petroleum Distillate	35 to 55	45.5
Polyethylene	3 to 10	8.0
Treated Nylon	0 to 5	2.5
Candelilla Wax	2 to 7	4.0
Beeswax	2 to 7	4.0
Lanolin	0 to 5	2.0
Genex 216	0 to 5	2.5
Floral 83	0 to 5	2.5
Bentone Gel SS71	10 to 20	16.5
Treated Iron Oxide Black	5 to 15	10.0
Treated Ultramarine Blue	0 to 5	2.0
Preservatives	0 to 2	0.5

In a closed mixing vessel, the liquid phase is headed to 75 to 80° C. The waxes are added and the mix is blended until uniform. The pigments are then added and mixture is blended until uniform. The product is cooled to 30° C then filled into a container.

While an illustrative embodiment of the invention has been described above, it is, of course, understood that various modifications will be apparent to those of ordinary skill in the art. Such modifications are within the spirit and scope of the invention

Claims

1. A powder or particulate material suitable for use in cosmetic products, said powder or material being coated with a trifluoro alkyl polysiloxane compound of the general formula



wherein m is an integer averaging from 1 to 1000;
n is 0 or 1; and
R_2 and R_3 is each hydrogen or an alkyl group of from 1 to 9 carbon atoms;
wherein at least one of R_2 and R_3 is a fluoroalkyl group substituted with from 1 to 5 fluorine atoms and having at least two carbon atoms if substituted with four or five fluorine atoms;
and wherein said fluoroalkyl groups are randomly or uniformly distributed in the polysiloxane structure.

2. A material according to claim 1, wherein said fluoroalkyl group includes a trifluoromethyl group.

3. A material according to claim 1 or 2, wherein m is an integer averaging from 500 to 1000.

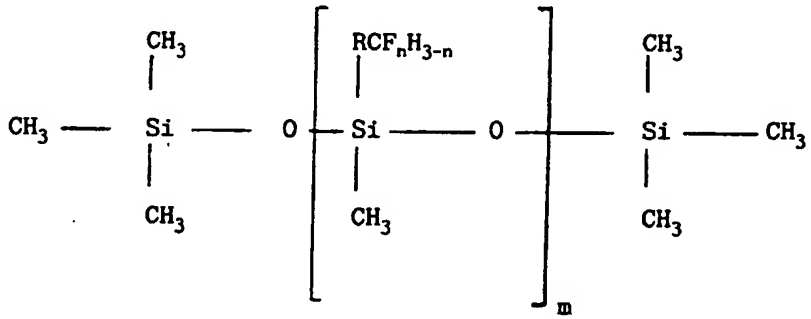
4. A material according to claim 3, wherein m is an integer averaging from 100 to 500.

5. A material according to any of the preceding claims, wherein R_2 and R_3 are alkyl groups comprising 1 to 4 carbon atoms.

6. A material according to any of the preceding claims, wherein said fluoropolysiloxane compound coating comprises 0.01 to 30.0 wt.% of the coated material.

7. A material according to any of the preceding claims, wherein m is an integer averaging from 1 to 100.

8. A powder or particulate material according to any of the preceding claims coated with a fluoroalkyl-polysiloxane compound of the general formula



m is an integer averaging from 1 to 500;
n is 0, 1, 2 or 3;
R is an alkyl group of 0 to 10 carbons;
and the fluoro groups may be randomly or uniformly disposed over the polysiloxane structure.

9. A cosmetic composition comprising a modified powder or particulate material according to any of the preceding claims.

10. An aqueous cosmetic dispersion containing a modified powder or microparticulate material according to any of the claims 1-8.

11. Use of a trifluoroalkylpolysiloxane compound as defined in any of claims 1-8 as coating for powders or particulate materials.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number EP 91202573.1

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	GB - A - 2 224 274 (POLA CHMICAL INDUSTRIES INC.) * Page 1, lines 1-6; page 5, lines 4-18; page 9, line 1 - page 11, line 2 * --	1-11	C 09 C 3/12 C 09 B 67/08 A 61 K 7/021
A	US - A - 4 732 931 (MAXSON) * Abstract; claims 1-3 * --	1,2	
A	US - A - 4 946 893 (SAITO et al.9 * Column 1, line 60 - column 2, line 16; column 2, lines 51-66 * ----	1,2,6, 8	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C 09 C C 09 B 67/00 A 61 K 7/00 C 08 L 83/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	09-12-1991	HAUSWIRTH

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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